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APPARATUS AND PRODUCT

## **SUBMISSION OF CERTIFIED PRIORITY DOCUMENT**

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Sir:

Applicants have claimed priority under 35 U.S.C. § 119 of Application No EP00204760.3 filed December 29, 2000 in Europe. In support of this claim, a certified copy of said application is submitted herewith.

No fee or certification is believed to be due for this submission. Should any fees be required, however, please charge such fees to Winston & Strawn LLP Deposit Account No. 50-1814.

Respectfully submitted,

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Die angehefteten Unterlagen stimmen mit der ursprünglich eingereichten Fassung der auf dem nächsten Blatt bezeichneten europäischen Patentanmeldung überein.

The attached documents are exact copies of the European patent application conformes à la version described on the following page, as originally filed.

Les documents fixés à cette attestation sont initialement déposée de la demande de brevet européen spécifiée à la page suivante.

Patentanmeldung Nr.

Patent application No. Demande de brevet nº

00204760.3

Der Präsident des Europäischen Patentamts; ACE EUROPETRO Auftrag

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Extrusion process, apparatus and product

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#### Extrusion process, apparatus and product

The present invention relates to an extrusion process for the manufacture of cooked cereals or dry pet food, an apparatus for carrying out the process and a product obtainable by the process.

US5997934 (Geromini et al.) discloses a process for the manufacture of cooked cereals or dry pet food which comprises preparing a mixture of water and a dry premix mainly comprising cereal flour or semolina, cooking the mixture and extruding it by pressing it through an extrusion die with the aid of a gear pump.

US5667833 (Juengling et al.) discloses a process and an equipment for manufacturing pasta which comprises preparing a mixture of cereal flour and water having a water content of 25-40%, kneading the mixture and extruding it by pressing it through an extrusion die with the aid of a gear pump.

The aim of the present invention is to provide an extrusion process and apparatus which maintain the starch's molecular structure in order to manufacture a product with properties close to those of a traditional roller-dried product and superior to those of a traditional cooked-extruded product.

As a matter of fact a traditional roller-dried product was not subjected to any mechanical stress during its whole manufacturing process, because a traditional roller-dried product was obtained by roller-drying a cooked slurry mainly comprising cereal flour and 40-45% water, the slurry having been cooked in a vat and the drying effect being obtained by pure heat transfer from the roller to the film of slurry applied on its surface.

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On the other hand, a traditional cooked-extruded product was subjected to high shear during its manufacturing process, because it was subjected to high pressure and friction while being cooked in the extruder.

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The process of Geromini et al. mentioned above in fact comprises extrusion-cooking the mixture of water and flour before pressing it through an extrusion die with the aid of a gear pump.

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On the contrary, the whole process of Juengling et al. mentioned above is carried out while maintaining the temperature of the mixture of water and flour or semolina below 55°C so that a raw pasta is obtained, without any gelatinization of the flour or semolina.

The process for the manufacture of cooked cereal products or dry pet food according to the present invention comprises preparing a mixture of water and a dry premix mainly comprising cereal flour or semolina, and pressing the mixture, with the aid of a gear pump, firstly through a heat exchanger wherein it is cooked and then through an extrusion die.

- Likewise, the apparatus for the manufacture of cooked cereal products or dry pet food according to the present invention comprises a mixing device, a gear pump, a heat exchanger and an extrusion die connected in series.
- Throughout the present specification and claims, the expression "mainly comprising cereal flour or semolina" means comprising at least 50% or preferably at least 70% of cereal flour or semolina.

Similarly, the expression "a mixture of water and a dry premix" means "either a mixture of water and dry premix or a mixture of steam, water and dry premix".

It has been observed, surprisingly, that it is possible with such a process and such an apparatus to maintain the starch's molecular structure in order to manufacture a cooked cereal product or dry pet food with properties close to those of a traditional roller-dried product and superior to those of a traditional cooked-extruded product.

This fact may especially be observed by comparing the respective gelatinization degrees and starch profiles.

1.5 The degree of gelatinization indicates the efficiency of the heat exchanger and how well the product was cooked. Typically, gelatinization of at least 85%, preferably of 90% or more indicates a sufficiently cooked cereal. The starch profile describes the repartition of the two 20 functional molecules of starch, amylose and amylopectin that directly influence important properties of the final product (e.g. viscosity, texture, solubility, and stickiness). Mechanical stress as applied in traditional single- and twin-screw extrusion mainly destroys the 25 amylopectin fraction thus reducing viscosity and increasing solubility and stickiness. This is why existing products made with low-shear technologies (roller drying, high moisture cooking) cannot be exactly copied by extrusion. Therefore, the present process aims to maintain the initial 30 starch profile by reducing shear. This is the precondition to obtaining products with properties similar to those of of roller dried or high moisture cooked products.

Thus, the product obtainable by the present process and apparatus preferably has a gelatinization degree of at least 85%, more preferably of 90% or more.

It also preferably has a starch profile characterized by respective proportions of 40-70% amylopectine, 5-22% intermediates and 15-35% amylose.

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In addition, the present product preferably has an expansion degree of from 1.5 to 10, more preferably of from 2 to 6.

- To carry out the present process for manufacturing cooked cereals, it is possible to prepare a dry premix comprising 70-88% of a flour or semolina of a cereal such as wheat, oat, corn and/or rice, and up to 20% of sugars such as sucrose and dextrose, to which other traditional
- ingredients may be added such as up to 2% of sodium chloride, up to 8% of oil or fat such as vegetable oil or fat or butter oil, up to 15% of modified starches such as modified corn or wheat starches, up to 10% of powdered milk or other protein source, up to a few % of cocoa powder, up to a few % of malt, up to 0.5% of mineral
  - powder, up to a few % of malt, up to 0.5% of mineral salts, up to 0.5% of vitamins, up to 2% antioxidants, and possibly even enzymes which can reduce the viscosity of the mixture to be prepared, for example.
- For manufacturing dry pet food, it is possible to prepare a dry premix comprising at least 50% of cereal flour, 25-35% of meat flour and animal by-products, 5-15% fat and tallow, up to 10% vegetable by-products and up to 6% vitamins and minerals, for example.

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For manufacturing either cooked cereals or dry pet food it is possible with the mixing device to prepare a mixture of water and the premix such that the mixture has a water content of 12-45%, preferably of 12-30% and more preferably of 12-22% by weight.

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The still un-cooked mixture is then pressed into the heat exchanger with the aid of a gear pump.

To this end the gear pump may be fed with this mixture in a continuous, steady way, while exerting on it a relatively low pressure of the order of from about 100 to 2000 kPa upstream of the gear pump, for example.

It is then possible to have the gear pump exerting a pressure of from 3000 to 25000, preferably from 4000 to 20000 and more preferably from 5000 to 16000 kPa on the mixture upstream of the heat exchanger.

Because the residence time in the gear pump is short and the mechanical energy input is low, the original raw materials may reach the heat exchanger without any major molecular degradation.

It is possible to cook this mixture by progressively
heating it to 90-180°C, preferably to 100-160°C for 2 to 15
min along the heat exchanger, for example.

The cooking in the heat exchanger occurs by mainly thermal heat transfer thus maintaining the original molecular structure. The properties of such a cooked product will be close to that of a traditional roller-dried product and superior to that of a traditional cooked-extruded product.

In order to press the mixture through said extrusion die, it is possible to maintain a pressure of from 2000 to 10000, preferably from 3500 to 9000 and more preferably from 4000 to 8000 kPa downstream of the heat exchanger and upstream of the die. The pressure loss accross the heat exchanger may be between 1000 and 20000, preferably between 1000 and 17000, and more preferably between 1000 and 12000 kPa.

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It is possible to cut the extruded rope of cooked mixture thus obtained into pellets or puffs just at the exit of the die with the aid of a cutting device, for example.

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As to the present apparatus, the mixing device may be a high speed blade mixer, a twin-screw intermeshing and corotating mixer or extruder, or a single-screw extruder, for example.

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The heat exchanger preferably is a static mixer heat exchanger. The static mixer may be of any adequate type such as the one disclosed in US5486049 (Boatman et al.) or US5538748 (Boatman et al.) or those equipping standard SULZER tubular heat exchanger such as SULZER's SMR DN80 type static mixer heat exchanger, for example.

The gear pump may comprise two toothed wheels, or toothed rollers which mesh with each other. The two rollers can rotate in a housing on axles situated in a plane perpendicular to the direction of a mixture stream. They can each rotate in the opposite direction, the teeth coming apart upstream and closing again downstream. The outer edge of the teeth can slide against a corresponding inner wall of said housing, thus defining intersticial spaces intended for transporting the mixture to be extruded downstream. The shape of the teeth may be especially designed to avoid as much possible any

shearing on the mixture.

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The extrusion die may comprise one or more extrusion conduits, especially cylindrical conduits, generally oriented in the same direction as that of a stream of cooked mixture to be extruded, for example. The shape of the extruded rope may be determined in particular by the shape of the outlet opening or orifice of said conduits.

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The cutting device may be a rotating knife, for example.

Embodiments of the present apparatus are described below by way of example only with reference to the accompanying drawings in which:

- Figure 1 is a schematic view of an embodiment of the present apparatus,
- Figure 2 is a schematic view of a heat exchanger static mixer which may be used in the embodiment of the apparatus represented in Figure 1, and
  - Figure 3 is a schematic view of another heat exchanger static mixer which may be used in an embodiment of the apparatus similar to the one represented in Figure 1.

As represented in Figure 1, the present apparatus comprises a mixing device 1, a gear pump 2, a heat exchanger 3/4, and an extrusion die 5 connected in series.

For carrying out the present process, the mixer 1 may be fed with the different components of the mixture through a hopper 6. At a downstream end of the mixer 1, a still uncooked but homogeneous mixture may be obtained. The gear pump 2 may be fed with this mixture in a continuous, steady way. The mixture may be pressed by the gear pump 2 firstly through the heat exchanger 3/4 (under the effect of a pressure p<sub>2</sub> exerted upstream of the heat exchanger), where it is cooked, and then through the die 5 (under the effect of a pressure p<sub>1</sub> maintained downstream of the heat exchanger and upstream of the die).

In the embodiment represented in Figure 2, the heat exchanger comprises two double jacketed, heatable barrels 3 and 4 filled with non heatable static mixers 7.

In the embodiment represented in Figure 3, the heat exchanger comprises one non heatable barrel 8 filled with a heatable static mixer 9.

The process and the product according to the present invention are described in greater detail in the examples presented below by way of illustration. The percentages are given therein by weight.

## 10 Example 1

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An apparatus corresponding to the embodiment represented in Figure 1 was used, which comprised a mixer in form of a twin screw side-feeder, a Vacorex 70/70 type MAAG gear pump, a heat exchanger as disclosed in US5486049 or US5538748 and represented in Figure 2, and a die in form of a steel plate provided with four cylindrical conduits each

A mixture of rice flour and water having a water content of 34% was prepared in the mixer which was fed with 48.2 kg/h of rice flour and 16.8 kg/h of water.

having a circular outlet opening 4 mm in diameter.

- The mixture was pressed by the gear pump through the heat exchanger under a pressure p<sub>2</sub> of 12200 kPa. The double jacketed barrels of the heat exchanger were heated to a temperature of 130°C with high pressure water. The mixture was thus cooked by having it heated to 117°C in a few min through the heat exchanger.
  - The pressure loss along the heat exchanger was 9800 kPa so that the mixture was then pressed through the die under a remaining pressure of 2400 kPa.
- 35 The product thus obtained had a gelatinization degree of 90.3%.

The product had a starch profile characterized by respective proportions of 59.4% amylopectine, 15.2% intermediates and 25.4% amylose.

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# Example 2

An apparatus corresponding to the embodiment represented in Figure 1 was used, which comprised a mixer in form of a twin screw side-feeder, a Vacorex 70/70 type MAAG gear pump, an SMR DN80 type SULZER static mixer heat exchanger such as represented in Figure 3, and a die in form of a steel plate provided with two cylindrical conduits each having a circular outlet opening 4 mm in diameter.

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A mixture of rice flour and water having a water content of 30% was prepared in the mixer which was fed with 51.1 kg/h of rice flour and 16.8 kg/h of water.

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The mixture was pressed by the gear pump through the heat exchanger under a pressure  $p_2$  of 12200 kPa. The static mixer of the heat exchanger were heated to a temperature of 130°C with high pressure water. The mixture was thus cooked by having it heated to 102°C in a few min through the heat exchanger.

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The pressure loss along the heat exchanger was 6900 kPa so that the mixture was then pressed through the die under a remaining pressure of 5300 kPa.

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The product thus obtained had a gelatinization degree of 92.1%.

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The product had a starch profile characterized by respective proportions of 65.0% amylopectine, 16.7% intermediates and 18.3% amylose.

# Example 3

The same apparatus was used as in Example 2.

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A mixture having a water content of 27% was prepared in the mixer which was fed with 51.6 kg/h of a premix comprising 85% rice flour and 15% sugar, 13.4 kg/h of water and 0.5 kg/h of oil.

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The mixture was pressed by the gear pump through the heat exchanger under a pressure  $p_2$  of 10900 kPa. The static mixer of the heat exchanger were heated to a temperature of 150°C with high pressure water. The mixture was thus cooked by having it heated to 118°C in a few min through the heat exchanger.

The pressure loss along the heat exchanger was 5500 kPa so that the mixture was then pressed through the die under a remaining pressure of 5400 kPa.

The product thus obtained had a gelatinization degree of 93.3%.

25 The product had a starch profile characterized by respective proportions of 57.9% amylopectine, 20.7% intermediates and 21.4% amylose.

#### Example 4

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The same apparatus was used as in Example 2.

A mixture having a water content of 20.3% was prepared in the mixer which was fed with 57.1 kg/h of a premix comprising 55% rice flour, 28% corn semolina, 13% sugar and malt and 4% cocoa powder, 7.9 kg/h of water and 2.3 kg/h of oil.

The mixture was pressed by the gear pump through the heat exchanger under a pressure p<sub>2</sub> of 13500 kPa. The static mixer of the heat exchanger were heated to a temperature of 150°C with high pressure water. The mixture was thus cooked by having it heated to 121°C in a few min through the heat exchanger.

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The pressure loss along the heat exchanger was 6000 kPa so that the mixture was then pressed through the die under a remaining pressure of 7500 kPa.

15 The product thus obtained had a gelatinization degree of 85% and an expansion degree of 1.5.

The product had a starch profile characterized by respective proportions of 51.6% amylopectine, 20.2% intermediates and 28.2% amylose.

#### Comparative example

For the purpose of comparison with the product obtained in
Example 4 a comparative sample was produced from the same
starting mixture but using a BC72H type CLEXTRAL twin-screw
extruder instead of the present apparatus.

The extruder was provided with a die in form of a steel

30 plate provided with six cylindrical conduits each having a circular outlet opening 3 mm in diameter.

The extruder was fed with about 3.5 times the amount of starting mixture with whom the mixer was fed in Example 4.

The extruder barrels were heated to achieve the same product temperature as in the SULZER heat exchanger.

The comparative product thus obtained had a gelatinization degree of 84%.

The comparative product had a starch profile characterized by respective proportions of 28.3% amylopectine, 48.3% intermediates and 23.4% amylose.

Thus the relatively low proportion of amylopectine remaining in the comparative product and the relatively high content in intermediates found in the comparative product stood in surprisingly stark contrast with the relatively high proportion of amylopectine maintained in the product of Example 4 and the relatively low content in intermediates found in the product of Example 4.

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#### Claims

- 1. A cooked cereal product or dry pet food obtainable by an extrusion process, which has properties close to those of a traditional roller-dried product and superior to those of a traditional cooked-extruded product.
- 2. A cooked cereal product or dry pet food according to claim 1, which has an expansion degree of from 1.5 to 10, preferably of from 2 to 6.
- 3. A cooked cereal product or dry pet food obtainable by an extrusion process, which has a gelatinization degree of at least 85%, preferably of 90% or more, and which has a starch profile characterized by respective proportions of 40-70% amylopectine, 5-22% intermediates and 15-35% amylose.
- 4. A cooked cereal product or dry pet food according to claim 3, which has an expansion degree of from 1.5 to 10, preferably of from 2 to 6.
  - 5. A process for the manufacture of cooked cereals or dry pet food, which comprises preparing a mixture of water and a dry premix mainly comprising cereal flour or semolina, and pressing the mixture, with the aid of a gear pump, firstly through a heat exchanger wherein it is cooked and then through an extrusion die.
- 6. A process as claimed in claim 5, which comprises preparing a mixture of water and the premix such that the mixture has a water content of 12-45%, preferably of 12-30% and more preferably of 12-22% by weight.
- 7. A process as claimed in claim 5, which comprises having the gear pump exerting a pressure of from 3000 to

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25000, preferably from 4000 to 20000 and more preferably from 5000 to 16000 kPa on the mixture upstream of the heat exchanger.

- 8. A process as claimed in claim 5, which comprises cooking 5 the mixture by progressively heating it to 90-180°C, preferably to 100-160°C for 2 to 15 min along the heat exchanger.
- 9. A process as claimed in claim 5, which comprises 10 maintaining a pressure of from 2000 to 10000, preferably from 3500 to 9000 and more preferably from 4000 to 8000 kPa downstream of the heat exchanger and upstream of the die.
- 15 10. An apparatus for carrying out a process according to any one of claims 3 to 7, which comprises a mixing device, a gear pump, a heat exchanger and an extrusion die connected in series.

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## Abstract

A process for the manufacture of cooked cereals or dry pet food which comprises preparing a mixture of water and a dry premix mainly comprising cereal flour or semolina, and pressing the mixture, with the aid of a gear pump, firstly through a heat exchanger wherein it is cooked and then through an extrusion die, an apparatus for carrying out the process and a product obtainable by the process.

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Figure 1





